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Superimposed high power impulse and middle frequency magnetron sputtering: Role of pulse duration and average power of middle frequency

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ABSTRACT

The low deposition rate of the high power impulse magnetron sputtering (HiPIMS) somewhat limits its commercial applications in thin film and coating industries. In this work, an effective way to improve the deposition rate of TiN film by superimposing the middle-frequency (MF) pulses during off-time of HiPIMS pulsing was discussed. Superimposition of HiPIMS and MF fabricated TiN films with relatively smooth surface. All samples were found to have a single phase TiN according to the chemical composition and crystallographic analyses. The highest film hardness of ~31 GPa, elastic modulus of 313 GPa, and a density of 5.35 g/cm^3 were obtained in the sample grown at a MF pulse duration of 150 μs in each superimposed cycle. The higher the deposition rate was achieved with longer MF pulse duration. This superimposition technique successfully improved the power-normalized deposition rate from 4.5 to 17.2 nm/kW·min without significantly declining the mechanical properties and adhesion quality of deposited films. The role of MF pulse duration and average MF power were further studied in this work. It can be concluded that the MF pulse duration played a more significant role and showed a dominant influence on the plasma characteristics and resulting properties of deposited TiN films.

1. Introduction

High density, corrosion- and wear-resistant hard coatings with excellent adhesion to steel substrates are in high demand for a wide range of applications in the metal forming, plastic molding, machining, automotive, petrochemical, food industries and decorative applications. High power impulse magnetron sputtering (HiPIMS) is a promising physical vapor deposition (PVD) technique, which produces high density plasma and ionized metal particles during sputtering [1]. HiPIMS technique is capable of offering target peak current, peak power density, and degree of ionization that are much higher than the conventional direct current (DC), radio frequency (RF), and middle frequency (MF) magnetron sputtering systems. Hence, the HiPIMS technique is reported to have a higher degree of target utilization and an improved thickness uniformity and densification of the deposited films as compared with those conventional ones [2]. Many works have been done to explore the potential applications of the HiPIMS [3–8].

Despite of these promising advantages, HiPIMS technique still suffers from its low deposition rate [7,9]. It becomes very essential to overcome this drawback. For instance, a hybrid HiPIMS and DC/RF co-deposition process has been demonstrated to show improvements on the deposition rate [10,11]. Another approach is by superimposing DC or MF powers into HiPIMS pulsing. This superimposition concept has also been shown to improve the deposition rate in pure Cr [12], reactive deposition of TiO_2 [13,14] and non-reactive deposition of pure Ti and Cu [15]. Superimposition concept in HiPIMS is very promising but its further application may be associated with many factors which require more research on the optimization of deposition parameters.

This study describes a superimposed HiPIMS and MF approach to reactively deposit TiN films in which the MF pulses are introduced during the off-time (t_{off}) of HiPIMS pulsing and compares their film properties with the sample in the case of single HiPIMS. In particular, we would like to investigate the role of MF pulse duration on the deposition process when the average powers of both HiPIMS and MF were fixed at 1000 W. The role of MF pulse duration was studied by varying the ratio of MF/HiPIMS on-time (t_{on}). In addition to MF pulse duration, we would also like to reveal the influence of average MF power on the deposition process, which is worth a study. The influence of average MF

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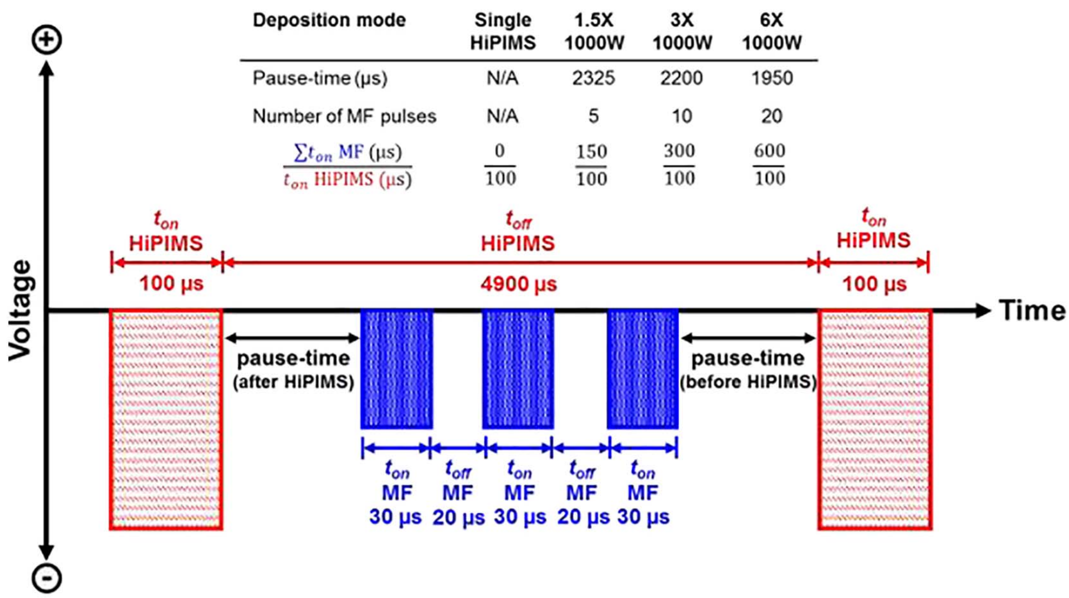

Fig. 1. Schematic drawing of pulsing configuration in a superimposed HiPIMS and MF system. The pause-time is adjusted to achieve different deposition modes.

power was studied by varying the average MF power from 1000 to 200 W while maintaining the average HiPIMS power at 1000 W. Therefore, two set of experiments were thus carried out.

2. Experimental procedures

2.1. Deposition techniques

Eight TiN films were reactively deposited on p-type Si (100) wafer and hardened AISI420 disc substrates by sputtering a rectangular Ti target (5 in. × 12 in.) under a gas mixture of Ar and N_2 in a chamber with the size of Φ 660 mm × 600 mm (LJ-UHV Technology Co., Ltd., Taiwan). Two deposition modes including single HiPIMS, and superimposed HiPIMS and MF were used in this work to fabricate eight films. For the superimposed HiPIMS and MF mode, the Ti target was powered by a HiPIMS power system (SIPP2000USB Dual, MELEC GmbH, Germany) consisting of two DC power sources, two entirely separated 4000 μF capacitor banks, HiPIMS and MF pulse generating devices.

For the single HiPIMS mode, the duty cycle and repetition frequency were maintained at 2% and 200 Hz, respectively. The pulsing configuration in the superimposed HiPIMS and MF mode is illustrated in Fig. 1. In the superimposed HiPIMS and MF mode, each superimposed cycle consists of one HiPIMS pulse and five to twenty MF pulses. The duty cycle and repetition frequency of HiPIMS and MF were set at 2%, 200 Hz and 60%, 20,000 Hz, respectively. The pulse t_{on} and t_{off} of HiPIMS were fixed at 100 and 4900 μs (duty cycle of 2%), respectively, while t_{on} and t_{off} of MF were set to be 30 and 20 μs (duty cycle of 60%), respectively.

To reveal the role of MF pulse duration, the ratio of total duration time, Σt_{on}, of MF to one duration time, t_{on}, of HiPIMS in one superimposed cycle was varied to be 150/100, 300/100, and 600/100, while the average powers of HiPIMS and MF were both fixed at 1000 W (defined as 1.5 × 1000 W, 3 × 1000 W, and 6 × 1000 W hereafter). For instance, 6 × 1000 W (six times) means that the total duration (Σt_{on}) of MF pulses was set to be six times longer than the t_{on} of HiPIMS pulsing in one cycle of superimposed HiPIMS-MF. In order to have the Σt_{on} of MF to be six times longer than the t_{on} of HiPIMS, the Σt_{on} of MF was set at 600 μs. To achieve this 600 μs, the time for introducing MF pulses before and after one HiPIMS pulse (defined as the pause-time) were adjusted to 1950 μs. In other words, there were 5, 10, and 20 cycles of MF pulsing in 1.5 × 1000 W, 3 × 1000 W, and 6 × 1000 W deposition modes, respectively, by adjusting the pause-time intervals. In this case, both HiPIMS and MF powers were run in the unipolar negative mode.

In addition to the pulse duration, the average MF power was also varied while the average HiPIMS power was fixed 1000 W. For this case, 6 × MF pulse duration was chosen and MF average power was raised from 200 to 1000 W, in which the samples were defined as 6 × 200 W, 6 × 400 W, 6 × 600 W, 6 × 800 W, and 6 × 1000 W hereafter, whereas the average HiPIMS power was fixed at 1000 W.

Before depositions, the Si wafer and hardened AISI420 disc substrates were cleaned with acetone and ethanol in ultrasonic cleaner. The substrates were then plasma etched for 15 min at a pressure of 1.3 Pa, an Ar gas flow rate of 40 sccm, and a DC substrate bias of − 800 V. Ti interlayer deposition was proceeded using single HiPIMS, and superimposed HiPIMS and MF modes for 20 and 5 min, respectively, at a pressure of 0.4 Pa, an Ar gas flow rate of 30 sccm, and a DC substrate bias of − 50 V. Finally, eight TiN films were grown at different deposition modes under an Ar and N_2 gas mixture ratio of 30 to 5. The sample designations and detailed deposition parameters are listed in Table 1.

2.2. Plasma characteristics

During depositions, a digital oscilloscope (Keysight InfiniiVision DSO-X 2024A, Agilent Technologies, USA) was employed to measure the voltage and current of Ti target. The ion-eroded area of the Ti target, 200 cm^2, was used to calculate the peak power density. An optical emission spectrometry (OES) system (PLASUS EMICON, Germany) was connected to the sputtering system to monitor the plasma emission spectra during deposition process. The setup of the optical spectrometry system was reported elsewhere [16].

2.3. TiN films characterizations

The surface morphology and roughness of TiN films were examined by a scanning electron microscope (SEM, JSM-6701, JEOL, Japan) and an atomic force microscope (AFM, DI-3100, Bruker, USA), respectively. The cross-sectional morphologies of TiN films were characterized by SEM and some selected samples were further examined by a transmission electron microscope (TEM, JEOL, JSM-2100, Japan). The chemical composition analysis was carried out using an field emission electron probe microanalyzer (FE-EPMA, JXA-8500F, JEOL, Japan). The crystallography of TiN films was evaluated by a glancing angle X-ray